

Section XIII: Identification of Nitrates

I. Introduction:

Samples are first screened by a color test with the Griess Reagent. For positive results, the sample is analyzed by GC/FID. For negative results, the sample is analyzed like any other unknown, by GC/FID analysis followed by GC/MS confirmation.

II. Reagents:

- A. 1% Sulfanilic Acid in 30% Acetic Acid (RGT#1).
- B. 1% N-1-Naphthyl-ethlenediamine in 30% Acetic Acid (RGT#2).
- C. Methanol (solvent)
- D. Alcohol Standard: solution of Isopropanol, n-propanol, Isobutanol, Butanol, Isoamyl, and Amyl.

III. Equipment:

- A. Test Tube (one for each color test needed)
- B. Glass pipettes
- C. 2 mL autosampler vials with Teflon caps
- D. GC/FID: HP 6890 or 7890A

IV. Procedure:

A. Griess Reagent Color Test:

1. Add 5 drops of Reagent #1 to a test tube.
2. Add 5 drops of Reagent #2 to the same test tube.
3. Add 1-2 drops of sample to mixture.
4. The color should turn a red or magenta color if Nitrite is present in the sample.
5. If this color appears, add 1-2 drops of the sample to an auto sampler vial and bring to volume with Methanol, then cap.

B. Analysis by GC/FID:

1. Place vials on autosampler tray with the following sequence: Standard, Blank, Sample.
2. The HP 5890 GC/FID conditions are:
Method: NITRITE.MTH
Oven:
Initial Temp: 35°C
Initial Time: 15 min.
Max. Temp: 120°C
Run Time: 15 min.

Inlet: (front injector only)
Temp: 150 °C
Gas Type: Helium
Detector: (front detector only)
Temp: 200 °C
Makeup Gas: Helium
Column:
HP Wax 15m x 0.25mm x 0.25um

V. Results:

- A. Report results in logbook, as well as the evidence cards. Be sure to include the date of analysis, result, and initials on the evidence cards.
- B. All reports generated from the instrument should be filed so they may be accessed at a later date, if necessary
- C. Butanol and Isobutanol are Class D controlled substances and Isoamyl and Amyl are Class E controlled substances.

GRIESS REAGENT

<u>NAME</u>	<u>FORMULA</u>	<u>CAS#</u>
Sulfanilic Acid (- 1% Sulfanilic Acid in 30% Acetic Acid)	C ₆ H ₇ NO ₃ S	121-57-3
1-Naphthylamine or	C ₁₀ H ₉ N	134-32-7
N-(1-Naphthyl)ethlenediamine (- 1% Naphthylamine in 30% Acetic Acid)	C ₁₂ H ₁₄ N ₂	1465-25-4

*NOTE: can use the N-1-Naphthylethlenediamine, it is more stable.